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## **FOREWORD**

This document is a final report covering work performed under Contract No. F49620-00-C-0036 from the Air Force Office of Scientific Research (AFOSR) Small Business Technology Transfer Research (STTR) program. The AFOSR contract monitor was Dr. Harold Weinstock. The Air Force project engineer was Dr. Paul Barnes of the Air Force Research Laboratory of the Wright Patterson Air Force Base, Dayton, OH. This report covers work conducted during the period 1 October 2000 to 30 September 2001. The research covered in this report was performed jointly by the Materials Laboratory of UES, Inc. and the Argonne National Laboratory (ANL). The key scientists from ANL involved in this project were Dr. U. (Balu) Balachandran, Director, HTSC Technology Program, and Dr. Beihai Ma, Staff Scientist. Besides the Principal Investigator and the author of this report, Dr. Rabi Bhattacharya, other key UES personnel who were actively involved in this project include: Mr. Maurice Massey, Mr. Howard Evans, and Mr. Robert Kerns. Ms. Jan Clark of UES, Inc. has provided excellent administrative support throughout this Phase I project.

## 1.0 INTRODUCTION AND SUMMARY

The work described in this report was funded by Air Force Office of Scientific Research (AFOSR) under a Phase I STTR project in partnership with Argonne National Laboratory (ANL). The work was initiated in October 2000, and the period of performance of this program was 12 months. The objective of the program was to develop textured buffer layer on metal substrates for the growth of high temperature superconductor such as  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$  (YBCO). While the original aim in Phase I was to develop in-plane textured MgO films on Hastelloy substrates (tapes) using Inclined Substrate Deposition (ISD) technique utilizing sputtering, the results of the work have shown more specifically that electron beam (e-beam) evaporation is more suitable for growing highly textured MgO films on static and moving Hastelloy tapes. Sputtering, on the other hand, has been found to be more suitable for growing a dense yttria-stabilized zirconia (YSZ) as a diffusion barrier layer between the Hastelloy and the ISD MgO layer. Also, sputtered YSZ and  $\text{CeO}_2$  grown epitaxially on ISD MgO resulted in an improved  $T_c$  and  $J_c$  characteristics of YBCO layers.

X-ray phi scan analysis has routinely provided good texture as represented by full width half maximum (FWHM) of MgO (002) peak within a range of  $12^\circ$  to  $14^\circ$  for the static sample and  $12^\circ$  to  $18^\circ$  for the moving sample. A reel-to-reel system was designed and built at UES for the ISD of MgO on moving tape inside an electron beam evaporation system. A 14" long tape was coated with textured MgO at a deposition rate of  $300\text{\AA}/\text{minute}$ . Bias sputtering was used to grow highly dense Yttria-Stabilized Zirconia (YSZ) films on Hastelloy substrates prior to the deposition of ISD MgO. This YSZ film has acted as an excellent diffusion barrier between MgO and Hastelloy. A multilayer architecture consisting of substrate Hastelloy/bias sputtered YSZ/ISD e-beam evaporated MgO/epitaxial e-beam evaporated MgO/epitaxial sputtered YSZ/epitaxial sputtered  $\text{CeO}_2$ /epitaxial laser ablated YBCO has been developed that showed reproducible high  $T_c$  of 89K and  $J_c$  in the range of  $1-2 \times 10^5 \text{ A/cm}^2$ . A schematic view of this structure is shown in Figure 1. ANL has performed the deposition of YBCO films by laser ablation and also the characterization of films for  $T_c$  and  $J_c$ .

780-800°C	PLD YBCO	0.5 - 1.0 $\mu\text{m}$
800° C	$\text{CeO}_2$ - Sputtered Epitaxy	0.02 $\mu\text{m}$
800° C	YSZ - Sputtered Epitaxy	0.5 $\mu\text{m}$
700° C	MgO - Ebeam Epitaxy	0.2 - 0.5 $\mu\text{m}$
Room Temp.	MgO - ISD Ebeam	1.5 - 2.0 $\mu\text{m}$
Room Temp.	YSZ - Bias Sputtered	0.5 - 1.0 $\mu\text{m}$
	Hastelloy C Substrate	

Figure 1. A Schematic View of the Multilayer Architecture of the Coated Conductor.

## 2.0 BACKGROUND

Most high-temperature superconducting thin films have been deposited successfully on single crystal substrates, in particular  $\text{SrTiO}_3$ . Although single crystal substrates promote the growth of oriented epitaxial films and are suitable for electronic applications, these substrate materials are not suitable for applications such as motors, generators, transmission cables and transformers for which long lengths of superconducting wires and tapes are needed. An important application of superconducting tapes could be in superconducting magnet technologies that support dipoles, quadrupoles, and higher order multipole corrector magnets for use in accelerators, storage rings, and charge particle beam transport system.

High  $T_C$  superconductors such as  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$  (YBCO) grown directly on polycrystalline metallic alloy substrates exhibit poor superconducting properties [1,2]. It has been demonstrated that a yttria-stabilized zirconia (YSZ) intermediate layer on single crystal Si and GaAs can yield YBCO films of higher quality [3-5]. Critical currents ( $J_c$ ) higher than  $10^6 \text{ A/cm}^2$  at 77 K have been reported for these films. However, YBCO films deposited under similar condition on polycrystalline metallic substrates with randomly oriented YSZ intermediate layers have shown two to three orders of magnitude lower  $J_c$  [6-8]. This discrepancy is attributed to the highly oriented growth of the YSZ layers on single crystal semiconductors because of lattice registry with these substrates. YSZ does not grow in such a highly textured orientation on polycrystalline metallic alloys. Recently, enhanced texturing of YSZ films has been reported for layers grown by an ion beam assisted deposition (IBAD) technique on highly polished Ni alloys [9-10]. It is suggested that grain alignment may be attributed to an energetic ion beam striking the growing film at an angle corresponding to the channeling direction of individual crystals in the film with the preferred orientation. The result is a net orientation of the polycrystalline YSZ film. Since the texturing of (100) YSZ evolves slowly, a thick film ( $>0.5 \mu\text{m}$ ) is required to achieve good in-plane alignment. This limits the application of YSZ as a buffer layer in actual manufacturing due to high processing time and cost. On the other hand, biaxially aligned MgO layers can be grown at a faster rate on inclined substrate without ion bombardment [11]. The advantages of this process are: (1) it is simple and fast, (2) it is a room temperature process, (3) texture is substrate independent, and (4) the process is easily scalable. The texture developed by this inclined substrate deposition (ISD) process is thought to be due to self-shadowing effect; i.e., grains with highest capture cross-section aligned with fast geometric growth direction overgrow grains that do not exhibit this alignment. ANL group has been pursuing this approach for past few years [12,13].

The other approach of developing textured MgO is based on IBAD, first demonstrated by Wang et al. at Stanford University. They have shown that in-plane textured (100) MgO can be grown in the form of a very thin layer,  $\sim 100 \text{ \AA}$ , by IBAD [14]. The mechanism of texturing of MgO is very different from that of IBAD (100) YSZ. The major difference stems from the fact that the preferred out-of-plane alignment for MgO is (100) direction as opposed to (111) for YSZ. Thus, the role of ion bombardment is to align the in-plane orientation. This simplifies the texturing process allowing the texture to appear at an early stage. However, the problem in this process is that the optimum thickness for the biaxial texture is around  $100 \text{ \AA}$ . Therefore, in-situ Reflection High Energy Electron Diffraction (RHEED) measurements are required for

monitoring and control of the development of texture. This prohibits easy scale up of the process.

### **3.0 TECHNICAL OBJECTIVES**

UES' initial Phase I technical approach was to develop a sputtering technique for the growth of in-plane (100) MgO on technical substrates suitable for manufacturing long conductor tapes. A specially designed biasing electrode was used in between substrate and the sputter target to achieve ion bombardment of the growing film along a desired direction. This approach has been successful in developing biaxially textured YSZ films on Hastelloy substrates. However, because of the constraints of growing textured MgO film by IBAD process, as described above, the approach of biased sputtering was not successful and was abandoned. Instead, the ISD process by e-beam evaporation was developed at UES (in collaboration with Argonne) for textured MgO growth, while the other layers for a multilayer architecture was grown by sputtering.

The specific objectives in Phase I were:

1. Deposition of MgO films on nickel alloy (Hastelloy) substrates using inclined substrate deposition. (UES)
2. Characterization of MgO films for texture as a function of deposition parameters and thickness of the films. (UES)
3. Set-up a reel-to-reel ISD process and demonstrate the deposition of textured MgO film on moving tape. (UES)
4. Develop a multilayer architecture incorporating the textured MgO layer for the growth of YBCO films. (UES)
5. Deposition of YBCO on selected multilayer MgO coated nickel alloy substrates. (ANL)
6.  $T_c$  and  $J_c$  characterizations of YBCO films. (ANL)

The research approach described in this report was designed to meet the objectives stated in the Phase I STTR solicitation and represent a direct technical response to the programmatic scope and work statement. It is the opinion of the author that this research has contributed to the development of a superior multilayer architecture by combining both e-beam evaporation and sputtering for the deposition of various layers.

### **4.0 RESEARCH WORK CARRIED OUT**

#### **4.1 EXPERIMENTAL DESCRIPTION**

ISD of MgO films were made by e-beam evaporation using the deposition system at UES. Mechanically polished Hastelloy C276 pieces of size 2 cm x 0.7 cm with a thickness of



0.15 mm were used as substrates for the MgO deposition. Some of these substrates were coated with bias sputtered YSZ. Typically, 2000 – 3000 Å thick YSZ films were deposited by radio frequency (RF) reactive magnetron sputtering using the dual magnetron sputtering system at UES. An Ar(80%) + O<sub>2</sub>(20%) gas mixture at a flow rate of 50 SCCM and a gas pressure of 2 mtorr were used for the deposition. RF power of 300W and a substrate bias voltage of –100 to –200V DC produced adherent and dense films of randomly oriented YSZ. Also, other coatings such as SiO<sub>2</sub> and TiN were used as diffusion barrier layers. SiO<sub>2</sub> was deposited by a reactive magnetron sputtering technique using silicon target. TiN coating was deposited by a unique filtered cathodic arc deposition technique. SiO<sub>2</sub> coating is amorphous and insulating, whereas TiN is crystalline with (111) preferred orientation and electrically conducting. A sketch of the ISD deposition set-up is shown in Figure 2. Small pieces of MgO crystals were used as the charge material for the e-beam source. The substrates were mounted at a height of about 30 cm from the e-beam source and inclined by an angle  $\alpha$  with respect to the substrate normal. The angle  $\alpha$  was chosen to be between 45° and 55°. A cylindrical collimator, 14 cm long and 4 cm diameter, was placed near the substrate. The deposition rate was measured by a quartz crystal thickness monitor placed near the substrate. The substrate was maintained near the room temperature. A deposition rate of about 2.5 Å/sec was used for the most of the depositions. However, experiments were conducted with higher deposition rates of up to 20 Å/sec to study the dependence of texture quality on the rate of deposition. Films of thickness in the range 2 to 2.5 microns were deposited. X-ray diffraction, Auger Electron Spectroscopy (AES) and Scanning Electron Microscopy (SEM) were used to characterize the MgO film on silicon substrate. The e-beam deposition of ISD MgO film was followed by an epitaxial MgO layer deposition by

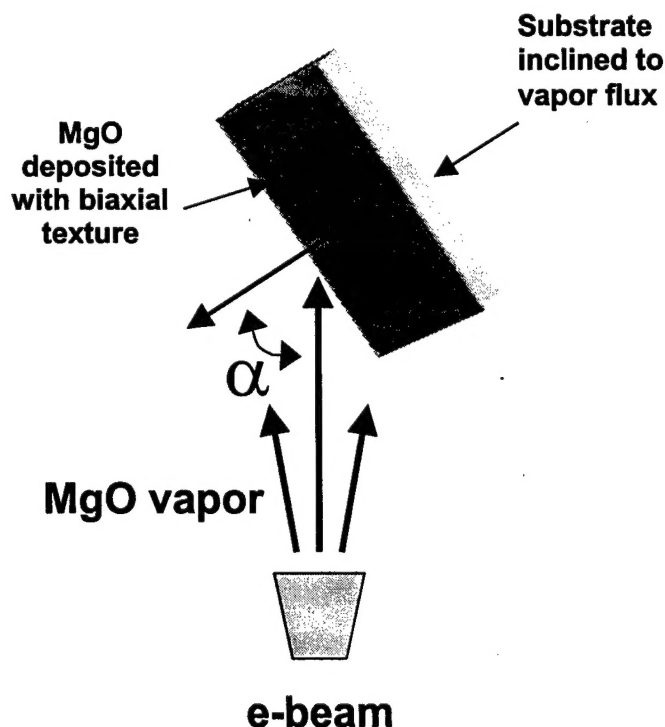


Figure 2. A Schematic View of the ISD Set-up.

e-beam evaporation in the same chamber. The deposition temperature for this film was 700°C. This film was grown with the deposition flux at normal incidence. An epitaxial MgO layer of thickness about 2000Å to 5000Å was deposited. This epitaxial MgO layer was necessary for smoothening the surface of the roof tile morphology of the ISD MgO film. A sputtered YSZ layer of thickness 5000Å was grown epitaxially at 800°C using magnetron sputtering. A deposition rate of 3 to 5 Å/sec was used. Finally, a 200Å thick CeO<sub>2</sub> layer was grown epitaxially by reactive magnetron sputtering. This multilayered film structure was characterized at every step by x-ray diffraction phi scan analysis for texture and epitaxy.

## 4.2 RESULTS

### Composition and Microstructure

Figure 3 shows the AES depth profile of the MgO film on silicon substrate. The relative Mg and O sensitivity factors were determined by using MgO crystals as a standard. It can be seen that Mg/O ratio is very close to 1.0 throughout the profile, indicating that the film is stoichiometric. Figure 4 shows the SEM photo of the MgO film surface. A roof-tile morphology with large gaps between the layers is clearly evident on the film surface. SEM of the fractured edge reveals the microstructure of the film in cross-section, Figure 5. A columnar microstructure is clearly evident.

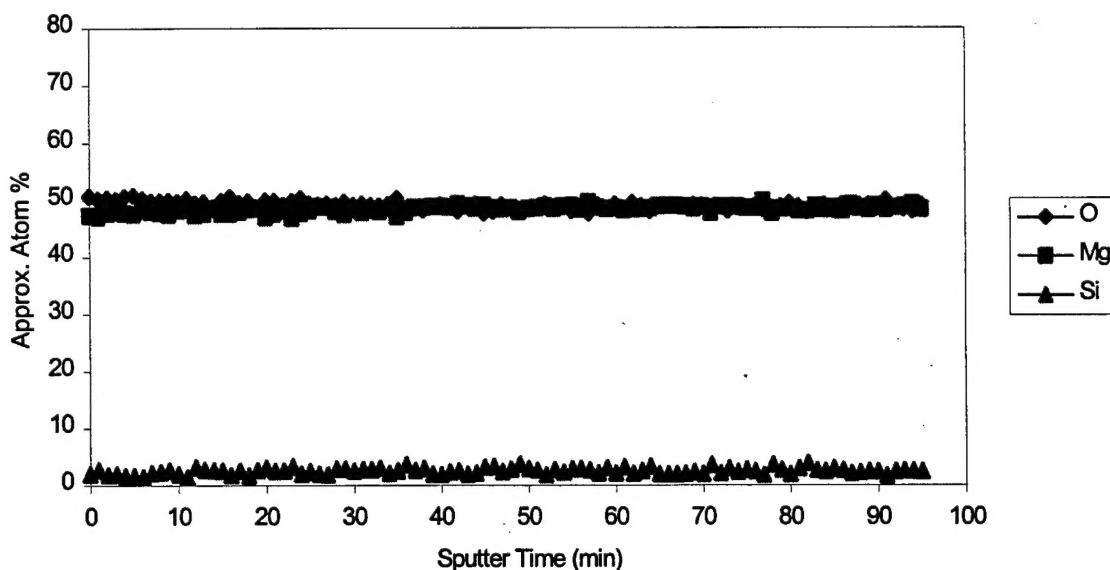


Figure 3. AES Depth Profile of the MgO Film on Si.

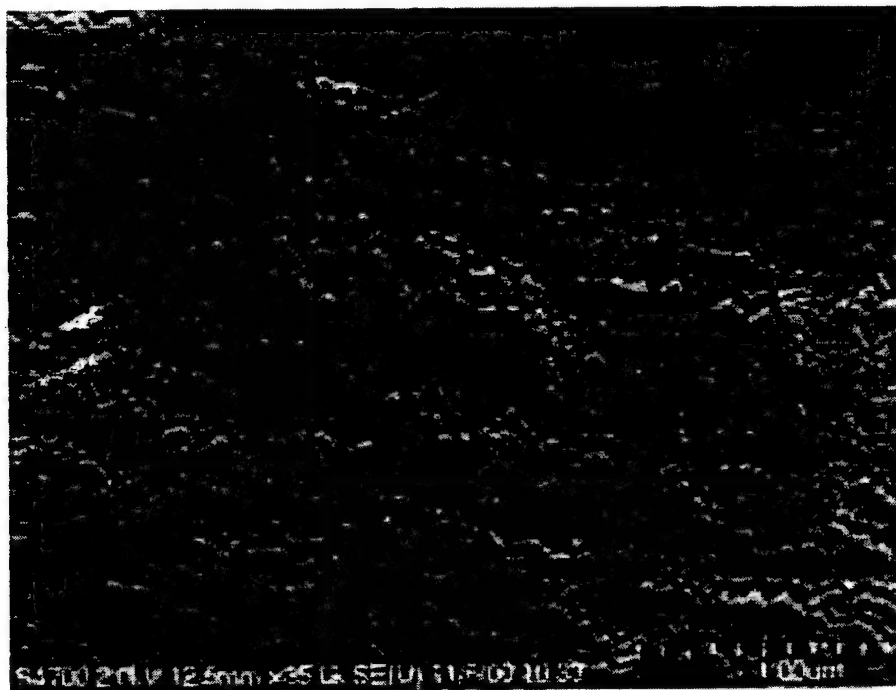


Figure 4. Scanning Electron Micrograph of the MgO Film Surface.

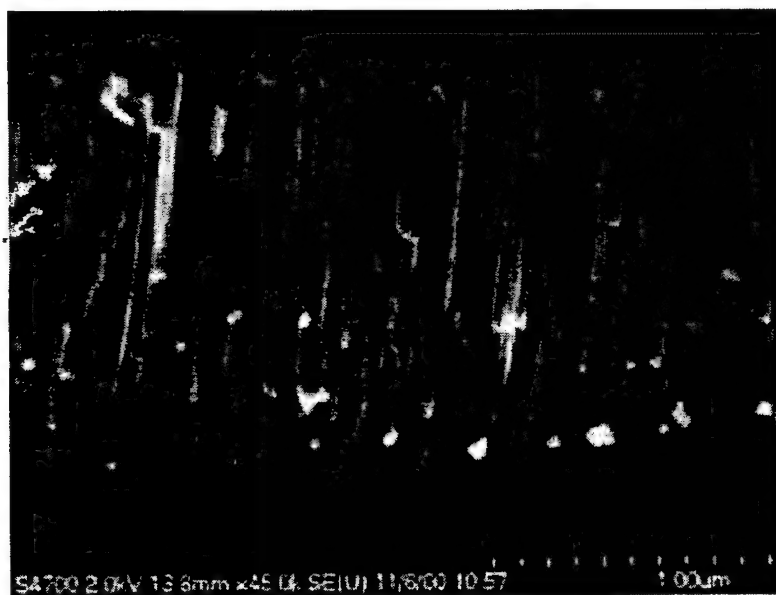


Figure 5. Cross-section Scanning Electron Micrograph of the MgO Film.

### Texture of ISD MgO Buffer Layer

All deposited films were characterized by x-ray diffraction phi-scan analysis. Figures 6 a,b,c show the results of pole figure analysis of samples 1, 2 and 3 that were deposited with  $\alpha = 54^\circ$  on SiO<sub>2</sub> coated (#1 and #2) and TiN coated (#3) Hastelloy. All three samples have good biaxial texture. MgO (002) plane has a tilt angle of  $30^\circ$ . FWHM of MgO (002) is about  $12^\circ$ . Figures 7 a,b,c show MgO (002) phi-scans of the above samples. FWHMs of phi-scan peaks agree well with that obtained from pole figure analysis. Figure 8 shows the phi-scan of a MgO film on a silicon substrate included in the same run with Hastelloy. Similar texture of the deposited MgO film reveals that the ISD process is not substrate dependent.

ANL has been working on a diffusion barrier layer for some time. Based on their suggestion, we have deposited Yttria Stabilized Zirconia (YSZ) films by biased sputtering on Hastelloy samples. ANL has used e-beam deposited YSZ films as a diffusion barrier layer and obtained good results. Bias-sputtered YSZ film should be denser than the e-beam deposited YSZ film thus providing a superior barrier to metal atoms. The bias sputtered YSZ films are polycrystalline in nature with random orientation. MgO films grown on the bias-sputtered YSZ film on Hastelloy showed good texture, as revealed from the phi scan in Figure 9.

### Texture of Epitaxial MgO on ISD MgO Layer

Figures 10 a and b show the phi scans of 1.5  $\mu\text{m}$  ISD MgO and a 0.5  $\mu\text{m}$  homo-epi MgO grown on top by using e-beam evaporation at a temperature of  $700^\circ\text{C}$ . This deposition was performed at normal incidence to the substrate surface. The epi grown layer shows slightly lower FWHM of the phi peak. Figures 11 a,b show the SEM micrograph of the surface and the cross-section of epi-grown MgO layer. These micrographs clearly show that the surface is smoother after the deposition of epi-grown MgO layer. The open pores of ISD MgO are no longer visible.

### Texture of Epitaxial YSZ/CeO<sub>2</sub> Layer

Figures 12 and 13 show the layer-by-layer pole figure and phi scan analysis of sputtered CeO<sub>2</sub>, sputtered YSZ and homo-epi MgO. Epitaxial growth of all of these layers are clearly revealed in these figures. The FWHMs of (002) and (220) of all these layers lie in the range of  $11$  to  $12.5^\circ$ .

### Deposition and Characterization of YBCO Layer

YBCO films were deposited by pulsed laser deposition (PLD) process at ANL. Substrate was attached to a heatable sample stage with Ag paste and heated to  $700$  to  $800^\circ\text{C}$  during deposition. The laser spot focused at the rotating target was  $2 \times 5 \text{ mm}^2$ , which provided an energy density of  $\approx 1 \text{ J/cm}^2$ . The distance between the target and the substrate was  $7 \text{ cm}$ . Base pressure of the chamber was  $2 \times 10^{-5} \text{ torr}$ . A high-purity oxygen flow of  $10 \text{ SCCM}$  was introduced into the chamber to achieve the desired operating pressure of  $0.1 - 0.3 \text{ torr}$ .

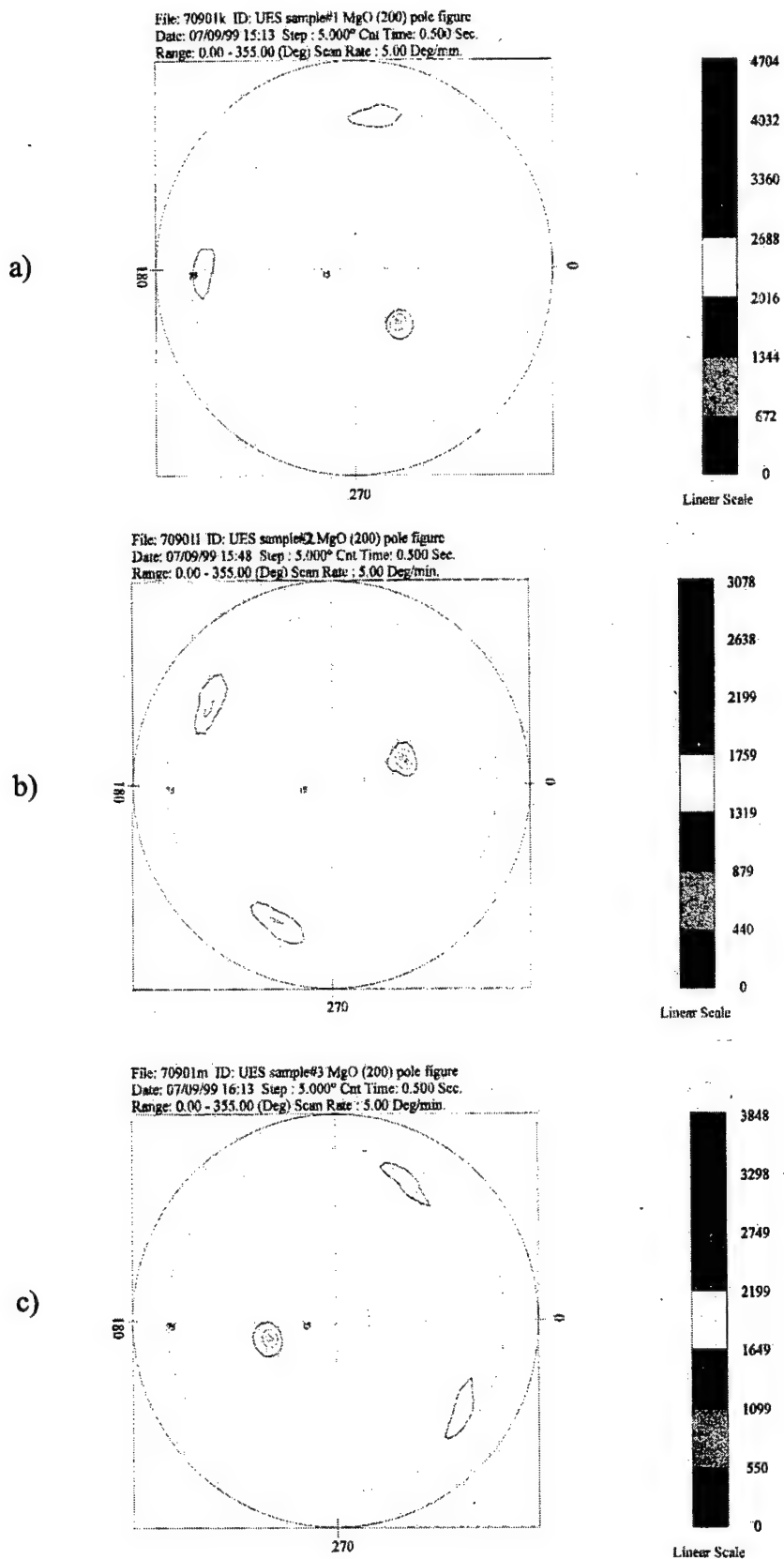


Figure 6. X-ray Pole Figure Analysis of MgO Films on Hastelloy: (a) and (b) SiO<sub>2</sub> Coated, (c) TiN Coated.

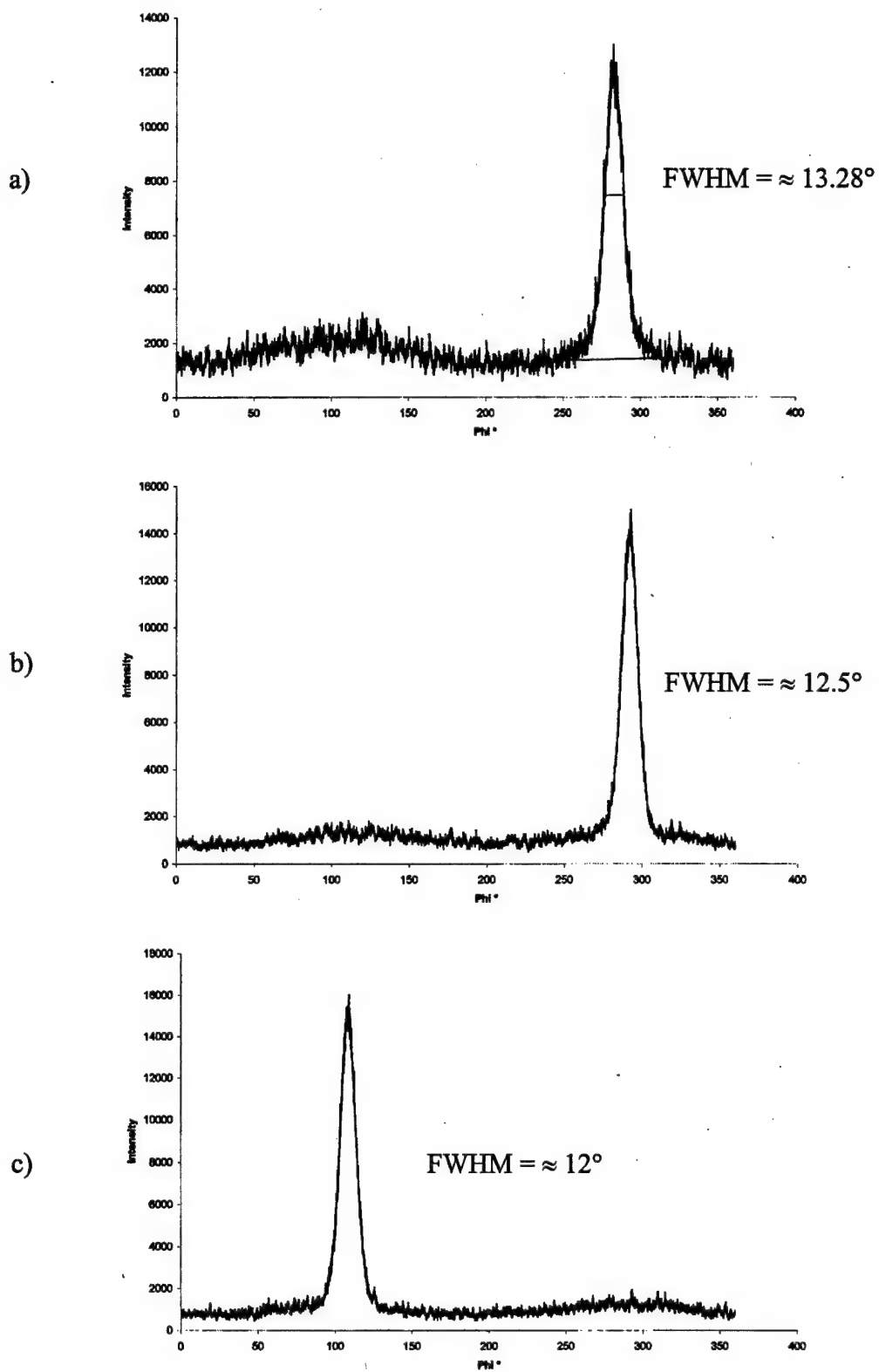


Figure 7. X-ray phi-scan of MgO (002): (a) and (b) SiO<sub>2</sub> Coated, (c) TiN Coated Hastelloy.

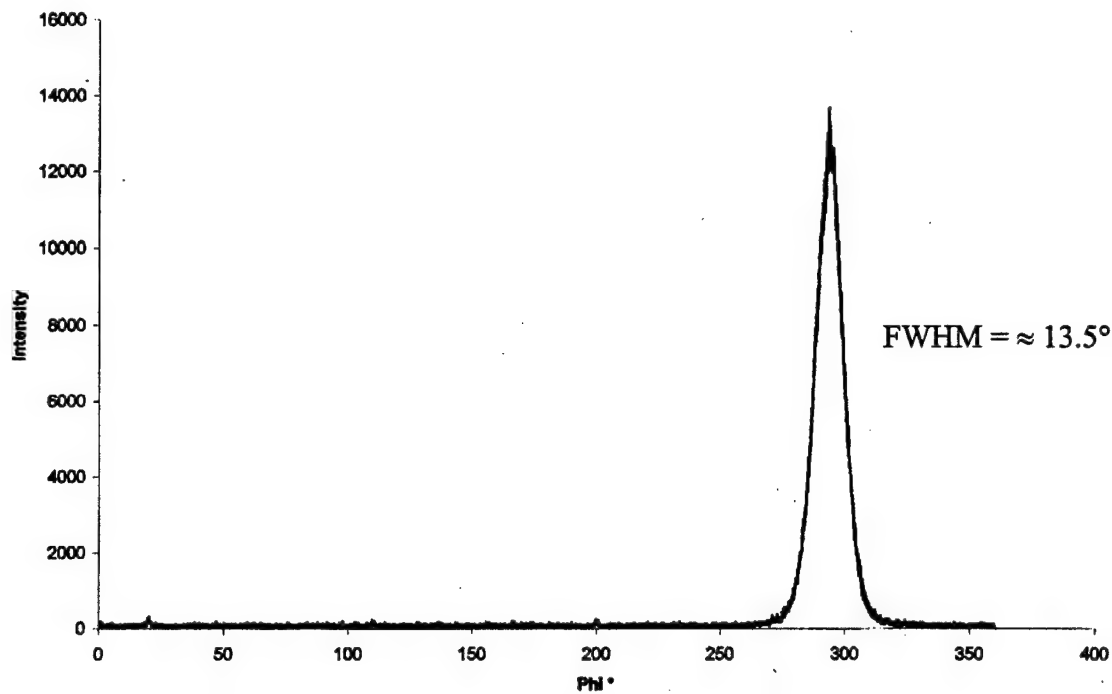


Figure 8. X-ray phi Scan of MgO (002) on Si Substrate.

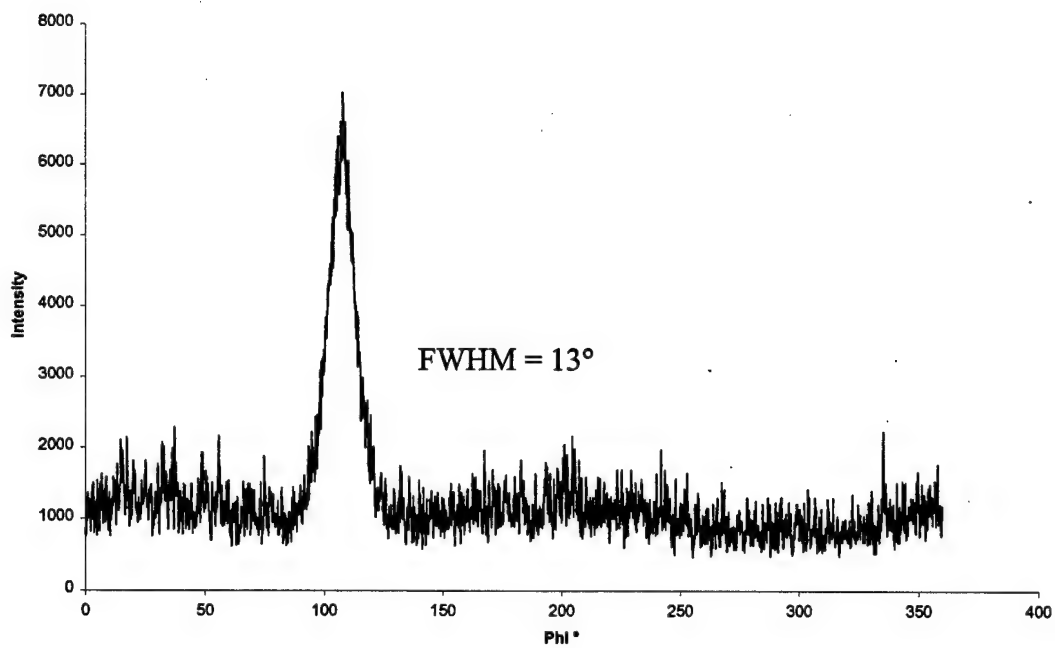
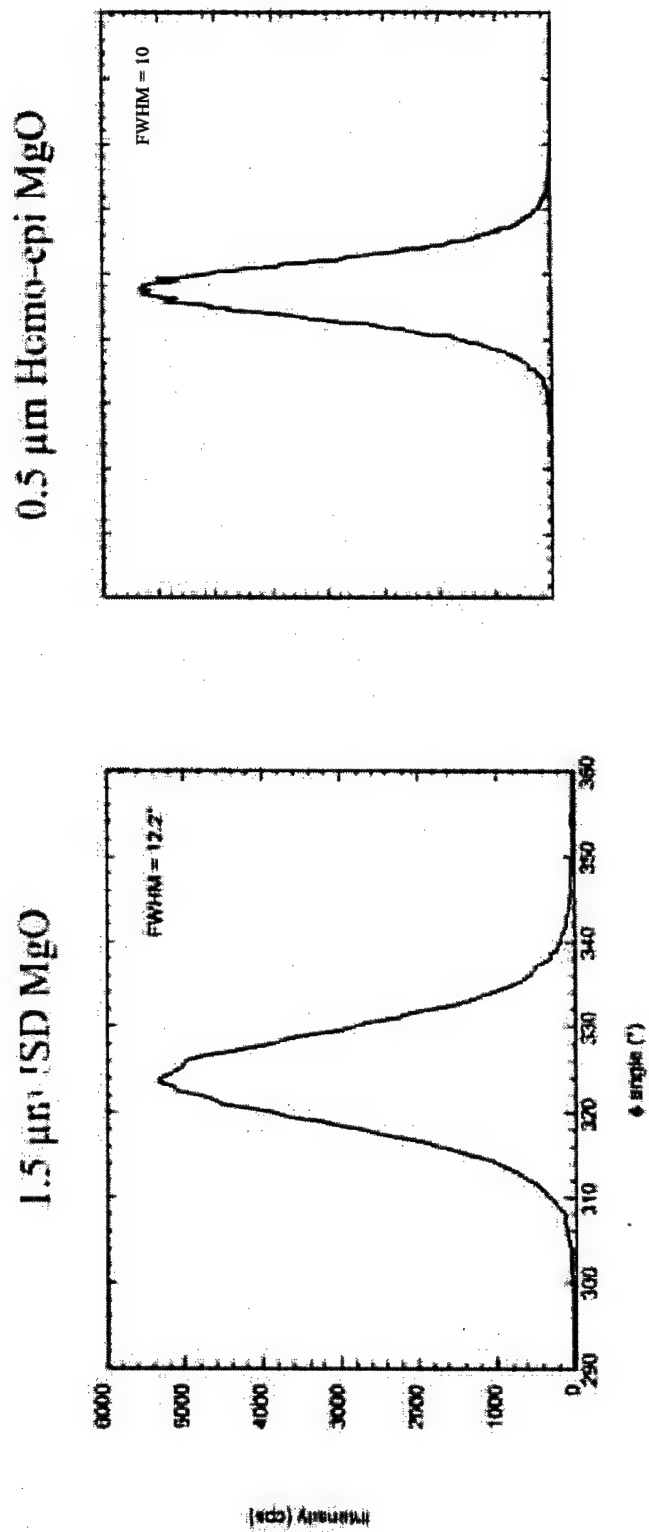


Figure 9. X-ray phi Scan of MgO (002) on Bias-sputtered YSZ Film on Hastelloy.



(a)

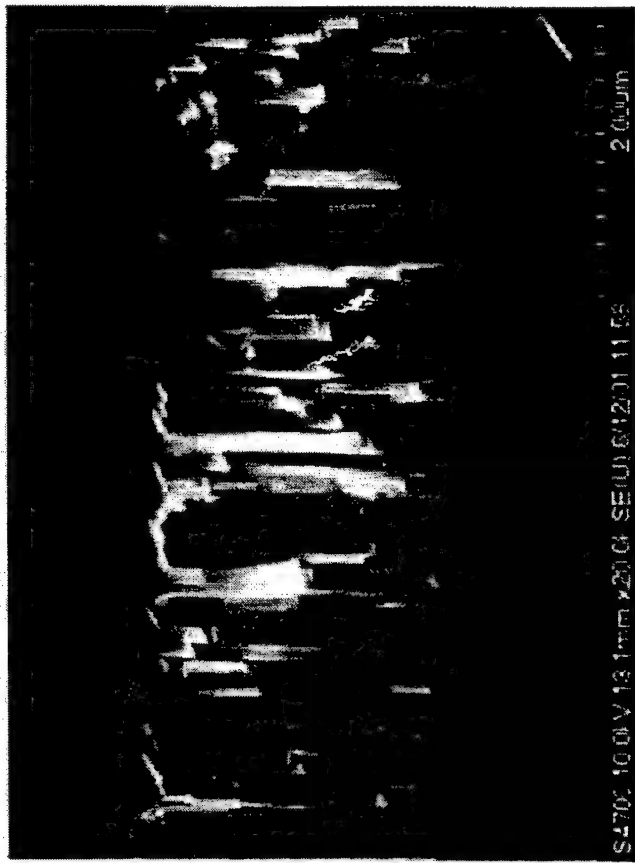
(b)

Figure 10. X-ray Phi Scan of (a) ISD MgO, (b) Homo-epi MgO Layers on YSZ Coated Hastelloy.





(a)



(b)

Figure 11. Scanning Electron Micrograph of the Epi MgO layer, (a) Surface, (b) Cross-section.

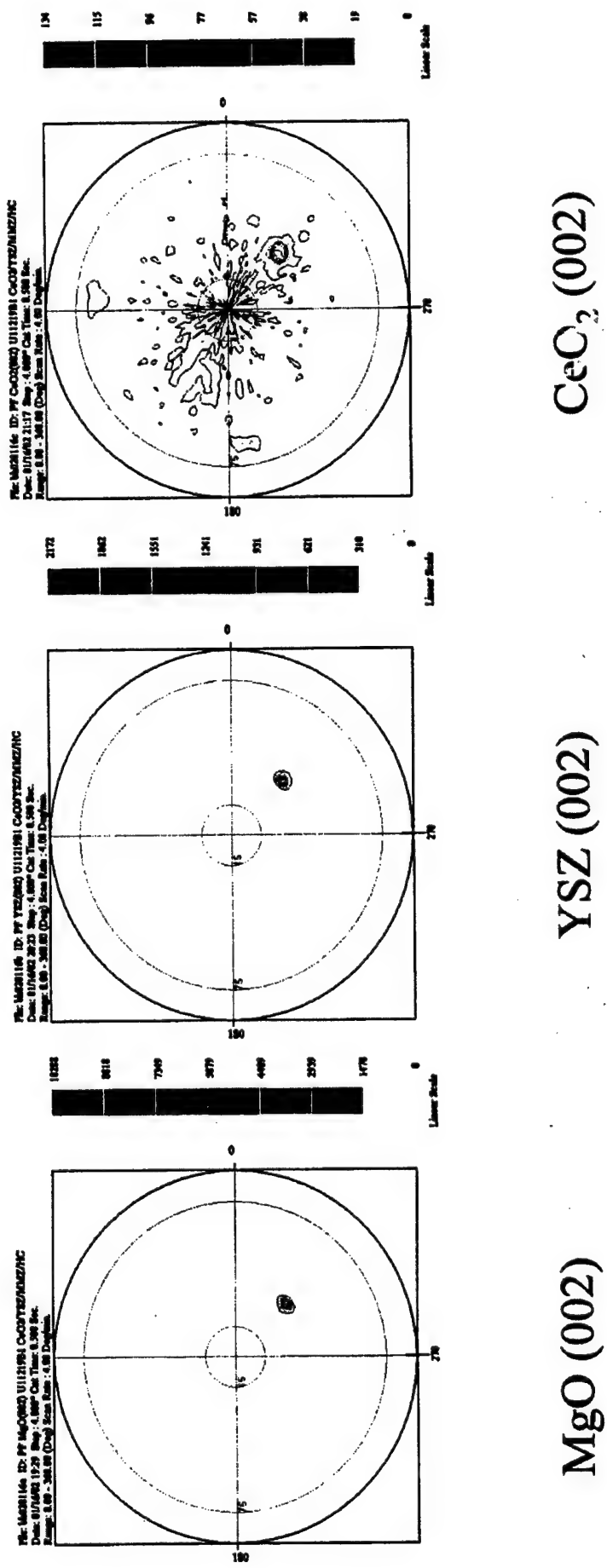


Figure 12. Layer-by-Layer Pole Figure Analysis of Sputtered CeO<sub>2</sub>, Sputtered YSZ, and Homo-epi MgO Layers.

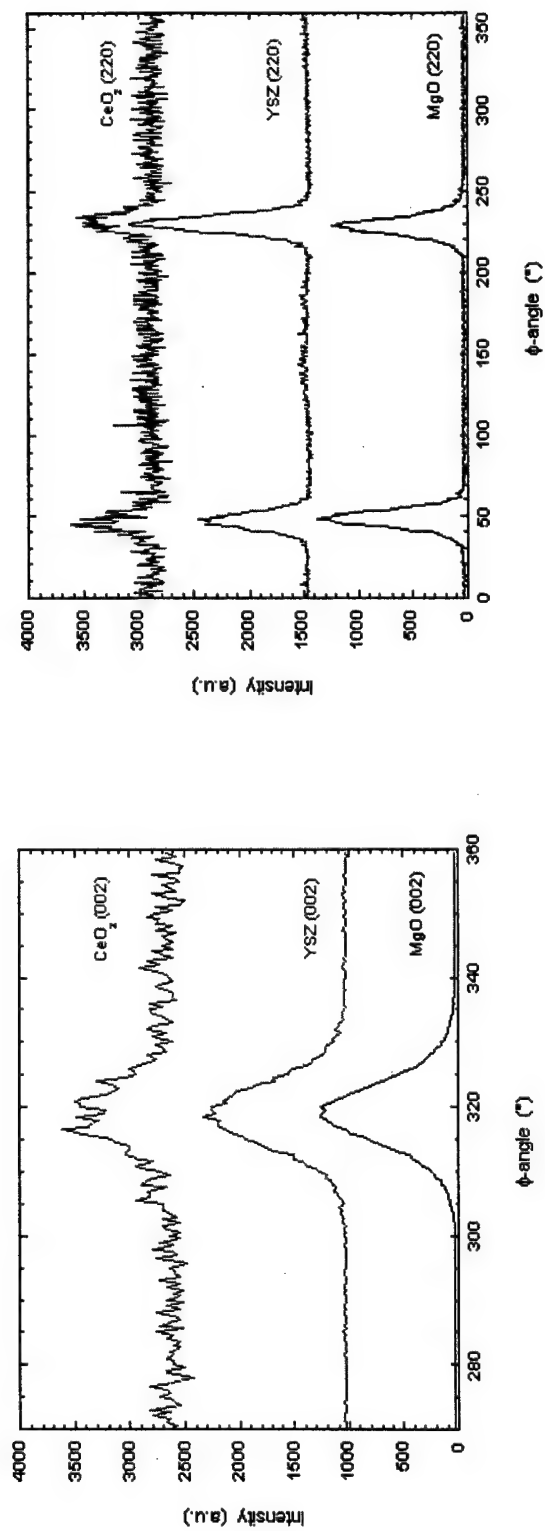


Figure 13. Layer-by-Layer X-ray Phi Scan Analysis of Sputtered  $\text{CeO}_2$ , Sputtered YSZ, and Hom-epi  $\text{MgO}$  Layers.

The superconducting critical transition temperature ( $T_c$ ) and critical current density ( $J_c$ ) were determined by the inductive method and confirmed by the transport method at 77 K in liquid nitrogen. The inductive test was used as a standard characterization tool to measure the superconducting properties of our YBCO films. Thin-film superconductor samples were placed between a primary and secondary coil pair with inner diameter of 1 mm and outer diameter of 5 mm. Alternating current was introduced to the primary coil and detected from the secondary coil by a lock-in amplifier (Stanford Research Systems SR830 DSP). Samples used for transport measurements were first coated with 2- $\mu$ m-thick silver by e-beam evaporation and then annealed in flowing high-purity oxygen at 400°C for 2 h. Typical samples used for four-probe transport measurement were 3-5 mm wide and 1 cm long.

Figure 14 shows the phi scan of the YBCO film deposited directly on an ISD textured MgO film. The figure shows the epitaxial growth of YBCO film with a FWHM of the phi scan = 10.2°. Note that YBCO (113) is parallel to MgO (220). This film, however, was not superconducting at the expected temperature of around 90K. This was because of diffusion of metal atoms into the YBCO film through the columnar, porous MgO buffer layer. YBCO films deposited on a multilayer architecture, as shown in Figure 1, using pulsed laser ablation (PLD) have shown the desired superconducting transition at about 89K. This is shown in Figure 15. Selected films have been evaluated for  $J_c$ . Values of  $J_c$  as high as  $2 \times 10^5$  A/cm<sup>2</sup> have been measured at 77K and in self-field.

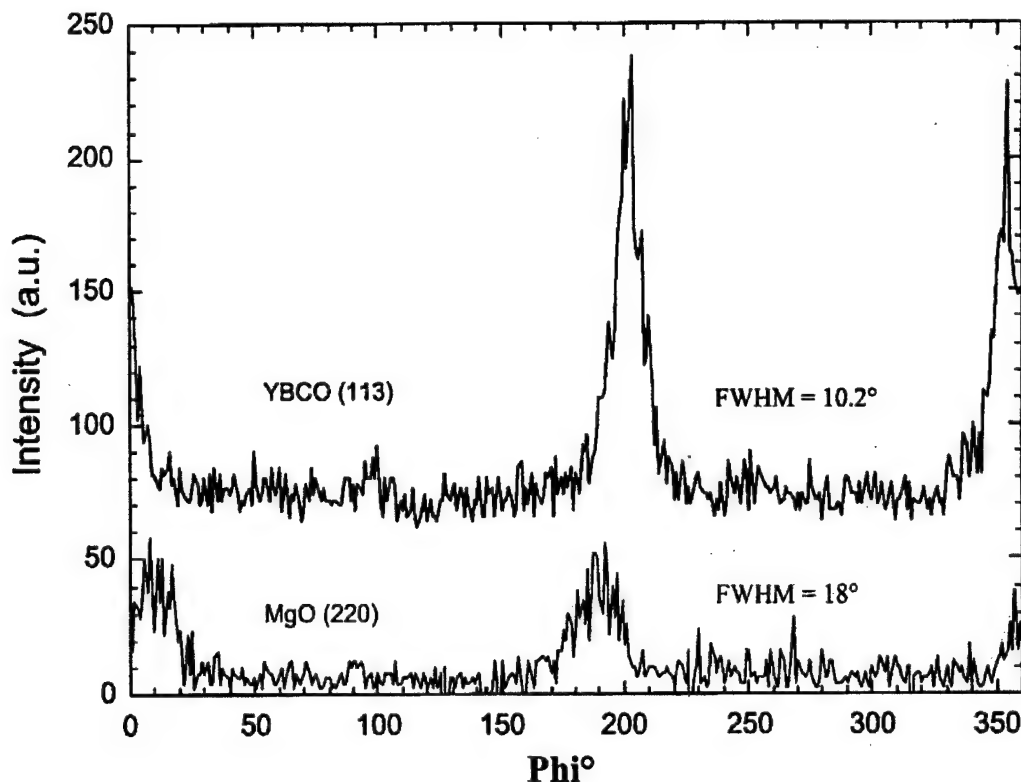


Figure 14. X-ray phi Scan of the YBCO Film Deposited Directly on an ISD MgO Film.

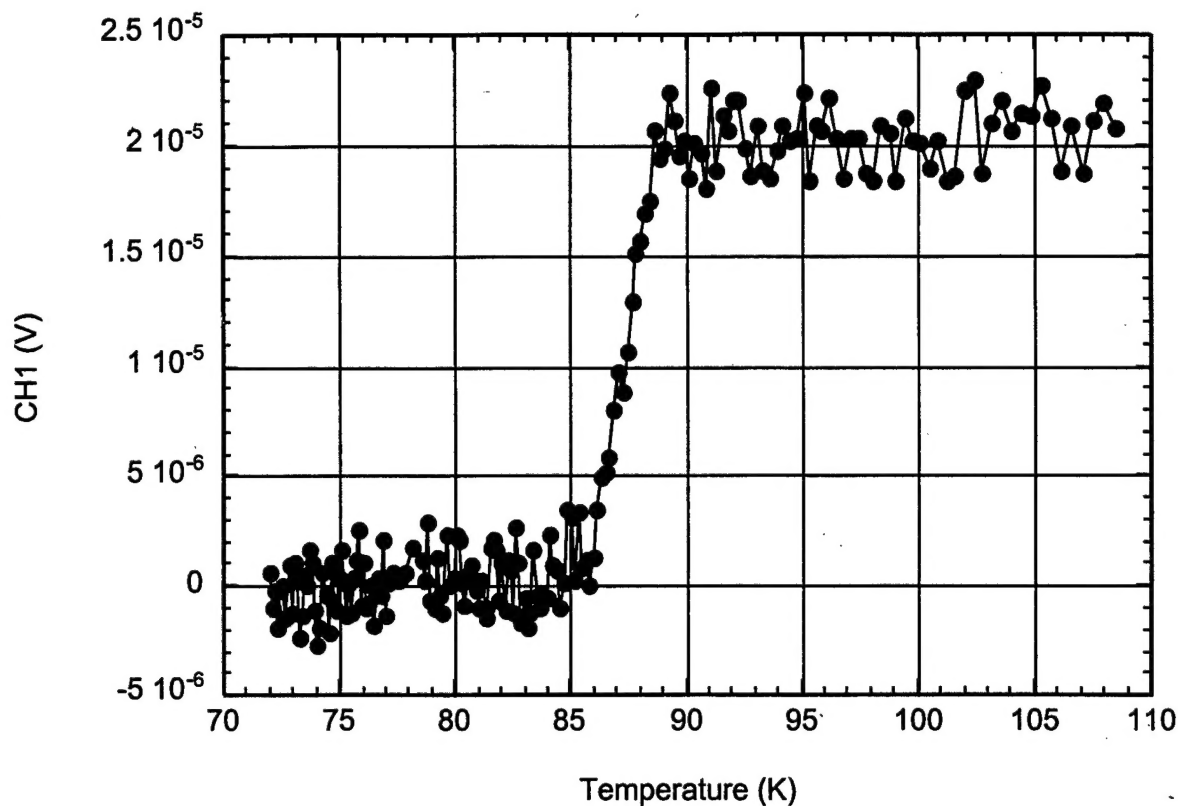


Figure 15. Plot of Induced Voltage (V) as a Function of Temperature (K) Demonstrating Superconducting Transition.

#### Reel-to-Reel Deposition of ISD MgO

We have also set up a reel-to-reel assembly for tape coating at UES' e-beam evaporation system. The photograph of the assembly is shown in Figure 16. This system is capable of producing up to 2 feet long coated tape. We have performed two runs so far using this system. A 12" long tape was coated at an inclination angle of  $50^\circ$ . The starting and the ending section of the tape (2 cm long sections) was analyzed for texture using x-ray phi scan. The beginning section showed a FWHM of  $12^\circ$ , while the ending section showed a FWHM of  $18^\circ$  of the phi scan.

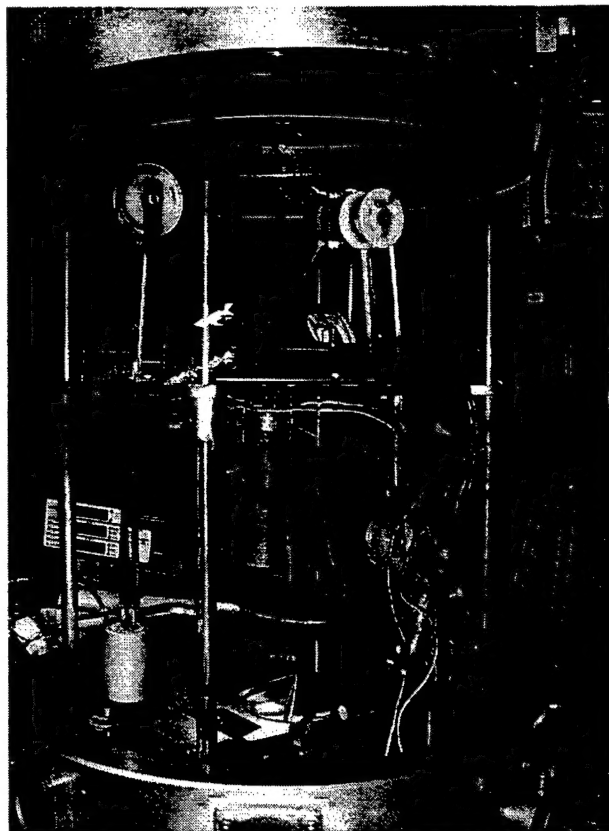


Figure 16. Photograph of Reel-to-Reel Assembly for ISD of MgO on Long Tapes.

## 5.0 CONCLUSIONS AND RECOMMENDATIONS

A twelve month technical effort was conducted to evaluate the feasibility of inclined substrate deposition of biaxially textured MgO layer on polycrystalline Hastelloy substrates. The project demonstrated the potential for combining e-beam and sputtering for growing a multilayer buffer architecture required for the epitaxial growth of YBCO layer.

### 5.1 CONCLUSIONS

The following specific conclusions can be drawn from the results of the Phase I research:

1. Inclined substrate deposition (ISD) by e-beam evaporation has been successfully used to produce biaxially-textured MgO films on polycrystalline Hastelloy substrates. The quality of texture as measured by the full-width-at-half-maximum (FWHM) of the MgO(002)  $\phi$  peak was in the range  $11^{\circ}$ - $14^{\circ}$ .
2. Deposition of textured MgO layer on a moving tape has been demonstrated by setting up a reel-to-reel tape moving assembly. A 14" long tape was coated with textured MgO with texture quality varying from  $12^{\circ}$  to  $18^{\circ}$ .

3. A multilayer architecture consisting of a Hastelloy substrate/sputtered YSZ layer/ISD MgO layer/epi MgO layer/sputtered epi YSZ/sputtered epi CeO<sub>2</sub> layer has been developed.
4. Yttrium Barium Copper Oxide layer grown on this multilayer architecture by pulsed laser deposition exhibited T<sub>c</sub> around 89K and J<sub>c</sub> in the range 1 – 2 x 10<sup>5</sup> A/cm<sup>2</sup>.

## 5.2 RECOMMENDATIONS

Based on the results of this project, the following recommendations are made:

1. The process developed in this project can be further optimized to obtain higher J<sub>c</sub>. This will require careful characterization and analysis of layer by layer growth of multilayer structure. Alternative materials for the diffusion barrier layer immediately adjacent to the metal substrate may be chosen. Also, the growth rate of individual layers should be optimized for scale up of the process for the production of long tapes.
2. The process should be scaled up for long tapes. This can be performed by modifying the UES' reel-to-reel sputter deposition system. One of the three sputter guns can be replaced with an electron gun for the ISD of MgO layer. The remaining sputter guns can be utilized for the in-situ depositions of multilayer architecture.

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